

INFLUENCE OF A BINDER LAYER ON THE RESPONSE TIME OF PRESSURE-SENSITIVE COATINGS

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Introduction

Pressure-sensitive coatings (PSP) offer a new highly promising means for visualizing and registering surface pressure in wind-tunnel facilities [1-5]. The method is based on dye luminescence quenching with oxygen. This technique has the following advantages: (1) it offers a non-destructive method for monitoring pressure distributions on model surfaces, (2) a very short time, about 2 – 10 s, is required for obtaining one image comprising one million pixels, (3) the method ensures a good measurement accuracy in transonic flows and is also applicable to low flow velocities, (4) the technique is capable of revealing flow features in real time (from luminescence intensity), (5) the technique offers a brilliant verification tool for numerical algorithms, (6) it permits quantitative measurements of pressure distributions in transonic and supersonic flows.

The emitted intensity and lifetime of dye are inversely proportional to local partial oxygen pressure and, since the oxygen content of air is constant, to air pressure. So to measure pressure distribution on the surface it is necessary to register the fluorescence/phosphorescence intensity or luminescence quenching time.

Registering steady-state fluorescence/phosphorescence intensity during continuous excitation is a simplest method for visualizing pressure with the help of a pressure-sensitive luminescent coating. However, dependence of measured signal on absolute excitation parameters (excitation intensity, number of excited phosphor molecules, film thickness, temperature) is a serious drawback of the method. To decrease these dependences PSP is formed on the base two luminophor (luminescence of the first luminophore is depends on pressure, luminescence of the second one do not depends), and to process optical data one usually use signal relation I/I_0 (where I_0 is intensity at ambient pressure and I is intensity at any pressure) [6, 7]. But it is not possible to overcome these disadvantages completely. Therefore pressure measuring using dye lifetime is perspective method [5, 8]. Particularly it is important when PSP is applied in short duration wind tunnel. In this case response time of coating on pressure change appears a restrictive factor of their using. Normally, preparation of a phosphor coating includes its encapsulation into a gas-permeable polymeric material and subsequent application (by sputtering) onto a surface to be studied. The response time of such coatings (which may amount to one second) is limited by oxygen diffusion into the polymer. Since the luminescence quenching requires collisions between molecules to occur or their coming into contact with each other, the limiting quenching rate depends on the diffusion velocity of migrating species in a given medium. To eliminate the effect of the rate of oxygen diffusion into the polymeric matrix on the time characteristics of PSP and reduce the coating response time to pressure variations, it is required to ensure an immediate contact between the luminescent species and the oxygen absorbed on the model surface. Adsorbents having open pores and a developed surface (open system) seem to be ideal systems for luminescent coating-assisted pressure measurement [4]. The oxygen-quenched luminescence of adsorbed phosphor molecules is a well-described phenomenon [9, 10]. At the present time, extensive studies of luminescent coatings prepared on commercially available quartz plates, anodized aluminum plates, or silica gel plates are under way [11-13]. Phosphor molecules applied onto such porous surfaces are immediately exposed to oxygen. The response time in this case is shorter than one millisecond, which is of extreme importance for conducting pressure measurements in short-duration wind-tunnel facilities.

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The present work describes an experimental study of pressure-sensitive luminescent coatings containing phosphors prepared on different substrates. Data are presented concerning measurements of luminescence intensity and luminescence decay kinetics at different pressures and temperatures.

Experiment

PSP's and their preparation procedure. The most important characteristics of luminescent materials are their electronic spectra, fluorescence intensity and fluorescence duration. The electronic spectra are caused by electron transitions between the ground state and excited states of phosphor molecules. Transitions from the ground state into excited ones are characterized by absorption spectrum, whereas reverse transitions by emission (luminescence) spectrum. The quantum yield (phosphor performance) is another important factor that determines the luminescence intensity. This parameter represents a ratio between the numbers of emitted and absorbed quanta. The absolute quantum yield does not depend on the output power of the light source and, as well as the absorption intensity, is determined by material structure. The quantum yield of many substances remains constant in a wide range of excitation wavelengths. Another important characteristic of a phosphor is its lifetime in an excited state after the excitation source was turned off (afterglow time). Depending on this time, either fluorescence ($10^{-9} - 10^{-7}$ s) or phosphorescence ($10^{-4} - 10^2$ s) is distinguished. For a practical use in aerodynamic research, some other phosphor characteristics are of importance: volatility, light resistance, solubility in certain polymer matrices, etc.

There are some organic substances which luminescence is quenching with oxygen. Ruthenium complexes, porpherin and pyrene refer to them. Luminescence lifetime of these phosphors is over some ns. In the study the phosphor was pressure sensitive modified pyrene. Pyrene attached to siloxane agglomerates is more stable and, unlike pure pyrene, does not volatile under pressure variation [14]. Luminophor coating was formed on the base of polymer and porous adsorbents. The film-forming polymer was SKTN polysiloxane resin. This substance is highly permeable for gases and has a short settling time for equilibrium oxygen concentration in the film (for a 20 μm -thick film, this time is about 1 sec). In the present study, the pressure-sensitive coating was obtained by sputtering 10% resin solution in toluene with controlled phosphor content. To obtain an "open system", we used as a substrate a silica gel/aluminum oxide mixture routinely used in chromatography. Due to its peculiar porous structure, silica gel has a developed surface area. The surface consists of siloxane bridges and silanol groups constituting binding sites for the phosphor. Powdered silica gel (mean particle size 20 or 60 μm) was mixed in water with aluminum oxide (1:1 by weight); afterwards, the resultant mixture was sputtered onto a surface to be studied. After a dry given to the substrate, the phosphor from the solution was applied onto it.

Experimental setup. The layout of the experimental facility is given in Fig. 1. Luminescence excitation was carried out by irradiation of 337 nm, impulse duration of 5 ns of lasers (1). The laser irradiation was supplied with mirror (3) into calibrating chamber (10) equipped with systems of heating, pumping out and letting gas. Pressure and temperature in the chamber were controlled.

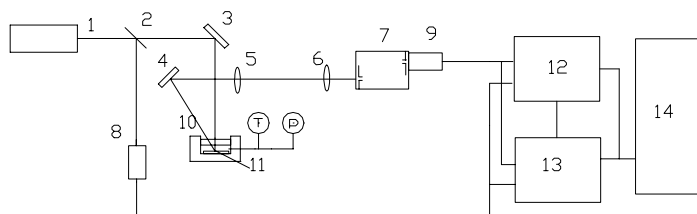


Fig. 1. Experimental setup.

The pressure varied from 0 to $2 \cdot 10^5$ Pa, temperature – from 20 to 100°C. Undercoat with applied PSP (11) was situated in the chamber. The spectral parameters of luminescence and decay kinetics were studied with the aid of double monochromator DMR-4 (7) and

either FEM-71 or FEM-87 (9) placed on an output slit. A part of radiation with the aid of beam splitter (2) came on photo-element FEC-22 (8) which voltage was a reference one and permitted to control the duration and intensity of the exciting radiation. Luminescence and laser temporal characteristics were registered with a high-speed digital oscillograph TDS-210 with a pass band of 60 MHz 90 (12). The integral characteristics of luminescence radiation on various wave length and of the laser were registered with the aid of ADC of controlling block CAMAC (13). The information from ADC and oscillograph was stored in computer (14) with special software.

Experimental results

Figure 2 shows the luminescence spectrum of modified pyrene. This spectrum was recorded for dye sputtered from toluene with dye concentration $5 \cdot 10^{-3}$ M/l. The maximum luminescence wavelength was 490 nm.

For PSP based on this dye encapsulated into a polysiloxane matrix ($5 \cdot 10^{-3}$ M of dye per one kg of polymer), the temperature (25–40°C) and pressure (0– 10^5 Pa) dependencies of the luminescence intensity and luminescence lifetime were measured. The luminescence intensity of the PSP based on CKTN-encapsulated modified pyrene is almost independent of temperature in the temperature range 20 to 80 °C (in vacuum). We carried out a stability test for a pressure-sensitive coating containing modified pyrene encapsulated into SKTN polysiloxane resin. Prior to measurements, the coating had been kept during ten days at room temperature. Figure 3 shows luminescence spectral characteristics of this coating. It is seen that the luminescence intensity and luminescence response time both remain unchanged.

A study of the lifetime τ of the PSP based on SKTN-encapsulated pyrene has shown that the time τ decreased from 400 to 100 ns as the pressure increased from 0 to 10^5 Pa (Fig. 4). The lifetime was determined at the moment when the intensity reached 90% of its initial value. The luminescence decay curve of SKTN-encapsulated pyrene can be well fitted with an exponential. The experimentally measured lifetimes versus pressure can be described well with a reciprocal function (Fig. 5). The scatter of the lifetimes for different samples at fixed pressure is well within 10%. Among the drawbacks of the coating is a high sensitivity of its luminescence kinetic characteristics to various contaminants in the pyrene and in the polymer. For gaining reliable experimental data, thorough pyrene purification and protection of coating from moisture are required. The pressure sensitivity of this coating is $0.15 \cdot 10^{-3}$ ns/Pa. The thickness of the coating in the range 5–25 μm was found to have no effect on the luminescence decay kinetics. The relaxation times of a 20- μm thick coating after letting-to-air and pumping were found to be shorter than 1 s.

To prepare a pressure-sensitive coating for measuring pressure in fast processes, we applied modified pyrene onto a porous coating with no «diffusion barrier». Figure 6 shows the kinetic curves of luminescence quenching of such PSC at various pressures. The luminescence decay kinetics is non-exponential both under the presence and under the absence of oxygen. The luminescence intensity decreases after a single evacuation. The latter phenomenon may be due to desorp-

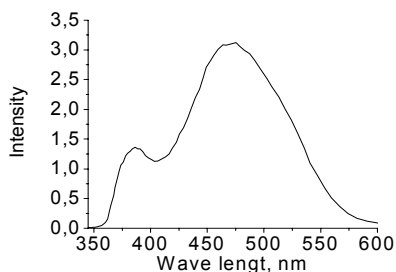


Fig. 2. Luminescence spectrum of modified pyrene.

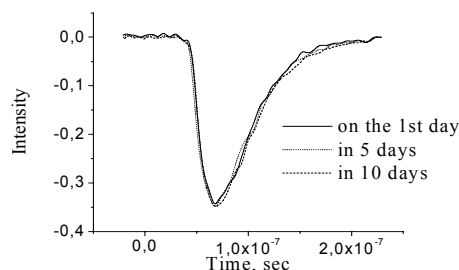


Fig. 3. Aging of PSP based SKTN-encapsulated modified pyrene.

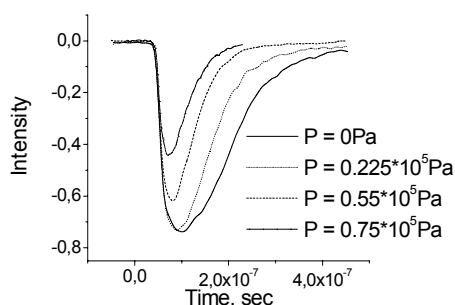


Fig. 4. Luminescence from SKTN-encapsulated pyrene versus pressure.

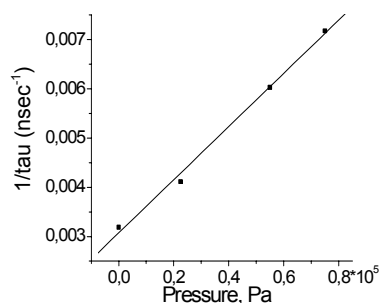


Fig. 5. Luminescence decay time of SKTN-encapsulated pyrene versus pressure.

tion of water molecules during pumping. In this case, water molecules are removed from the surface, oxygen diffuses more easily into the paint, and the quenching efficiency increases [15]. This assumption is confirmed by the following test. In one sample, the phosphor was applied onto a porous silica gel surface pre-calcined at 200°C for one hour, whereas in another sample the phosphor was applied onto a surface that was given no heat treatment. The luminescence intensity from the non-heat-treated sample turned out to be higher, showing that the luminescence intensity decreases during dehydration. The particle size of silica gel particles substantially affects coating-surface quality. A coating with 20-μm silica gel particles had a more even (flat) uniform surface, but simultaneously displayed a lower sensitivity to pressure variations. The response time of all samples was 45 ns under atmospheric conditions and 135 ns in vacuum, which is far shorter than that for a polymer-encapsulated PSP.

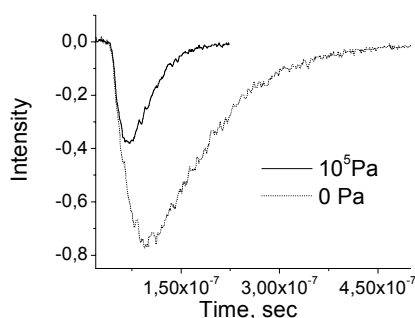


Fig. 6. Luminescence from pyrene applied on a silica gel substrate versus pressure.

Conclusions

Spectral characteristics and kinetic parameters of oxygen-quenched luminescence of different PSP have been measured. A comparison of various coatings prepared on a polymeric base or on a porous surface was made in terms of modified-pyrene luminescence intensity and luminescence lifetime. The data obtained in present experiments luminescence have shown the influence of binder layer on intensity and lifetime of PSP. When modified pyrene applied onto porous surfaces (silica) the response time of this coating is considerably short than the response time of polymer-encapsulated pyrene.

The pressure sensitivity of PSP on the of base of pyrene and porous binder layer is $0.15 \cdot 10^{-3}$ ns /Pa. But it is necessary to note there is a problem of insufficient adhesion of PSP coating to surface under study.

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